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# **VARIOUS METHODS OF PROCESSING SILICON-BASED COMPOSITES FOR ARMOR APPLICATIONS**

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July 1976

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## ABSTRACT

Silicon-boron carbide compositions containing 10 to 20 wt%  $B_4C$  were fabricated for ballistic evaluation against fragments and small arms projectiles. Fabrication was carried out by a variety of processing techniques to evaluate potential process technology. Among all the techniques investigated, hot pressing and liquid-phase sintering are the most promising with respect to density, microstructure, ease of fabrication, and yield rate. The hot-pressing parameters are established to be 1370 C for Si-10 wt%  $B_4C$  and 1440 C for Si-20 wt%  $B_4C$ , while the corresponding processing parameters by the liquid-phase sintering technique are 1410 C and 1520 C. For scale-up operation, the most appropriate composition was found to be Si-20 wt%  $B_4C$  due to minimum shape changes in the finished specimens. Cold forming of flat plates and complex shapes had been successfully achieved by a slip casting process. Ballistic evaluation indicated that low density Si-20 wt%  $B_4C$  compositions provide protection against fragments and small arms ball projectiles comparable to that of hot-pressed boron carbide.

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## INTRODUCTION

The materials which have been used or considered for use as personnel armor for ground troops may be grouped as (1) fabrics and felts; (2) metals; (3) fiber-reinforced plastics; and (4) ceramic-faced composites. Of these, only the ceramic-faced composites are effective in defeating both AP and small arms projectile threats at acceptable weights; the others are all fragment-protective materials. Ceramic materials which have been evaluated for or used in personnel armor items are high boron compounds such as  $B_4C$ ,<sup>1-4</sup>  $B_6Si$ ,<sup>5</sup> and  $CaB_6$  for protection against armor-piercing (AP) projectiles,<sup>6</sup> and  $Al_2O_3$ -GRP and  $SiC$ -GRP for small arms protection. However, even boron carbide, which is the most ballistically efficient of these materials due to its combination of low mass density and high modulus, will not meet the reduced weight requirements for future systems. Thus the best possibility for improving ceramic-faced armor against small arms projectiles and fragments is to explore materials and minerals whose theoretical densities are less than 2.3 g/cc. Due to the different nature of threats from small arms projectiles and fragments (as compared with AP), it was anticipated that the hardness requirement could be relaxed to approximately 1200 to 1400 Knoop. The materials selection criteria were, therefore, for very low density (2.3 g/cc or less), low cost potential, with a minimum of 1200 Knoop hardness, and ease of ultimate fabricability. These immediately impose restrictions in materials selection, limited to selected glass ceramics and very few metals (Si and Mg). This study focused on the evaluation of the potential for producibility of metallic silicon-based materials for personnel armor applicability. Silicon has a calculated density of 2.33 g/cc, is relatively cheap, plentiful, and fabricable, but has a hardness about 800 to 900  $K_h(100)$ . Therefore, the major technical objective of this program was to improve the hardness of Si by synthesizing new compositions, or by alloying additions to Si, or dispersion hardening of a Si matrix. This report emphasizes the fabrication of Si-based material systems for small arms protection requirements. These systems were generally  $B_4C$  dispersion-hardened silicon metal, fabricated by a variety of techniques.

## EXPERIMENTAL PROCEDURES

### A. Powder Characterization and Preparation

Silicon powder (99%) was obtained from Union Carbide, while boron carbide (99.7%) was obtained from Walker Chemicals. Tables 1 and 2 show the results of analyses carried out on Si and  $B_4C$  powders. Silicon powder with  $B_4C$  additions varying between 10 and 30 wt% were mixed in a high speed Blendex mixer. The dry mixing was carried out intermittently for a total period of one-half hour.

1. HANSEN, J. V. E. *Boron Carbide Body Armor*. Metal Progress, February 1969.
2. Limited Production Purchase Description for Body Armor, Small Arms Protective, (.30 cal. ball) Variable, Front and Back Plates, U. S. Army Natick Res. Lab. LP/P. DES 2-71, January 1971.
3. Limited Production Purchase Description for Body Armor, Small Arms Protective, Aircrewman, U. S. Army Natick Res. Lab. LP/P. DES 5-71, March 1971.
4. SEMPLE, C. W. *Ceramic Composite Armors (U)*. Army Materials and Mechanics Research Center, AMRA TR 65-26, October 1965 (Secret Report).
5. DUTTA, S. K., GAZZA, G. E., and RODERICK, D. J. *Investigation of Silicon Hexaboride as a Lightweight Armor Material (U)*. Army Materials and Mechanics Research Center, AMMRC TR 71-46, November 1971 (Confidential Report).
6. DUTTA, S. K. *Ballistic Properties of Hot-Pressed  $CaB_6$  (U)*. Army Materials and Mechanics Research Center, AMMRC TR 72-20, June 1972 (Confidential Report).



Table 1. SEMI-QUANTITATIVE SPECTROCHEMICAL ANALYSIS OF SILICON POWDER\*

Constituent	Content
Al	0.01 - 0.1 wt%
Fe	1.00 wt%
Mn	0.1 wt%
Mg	0.01 wt%
Cr	100 ppm
Ni	10-100 ppm
Ti	10-100 ppm
Ca	10 ppm
W	ND
Pb	ND
Co	ND

\*Analysis performed by Chemistry Lab, AMMRC

Table 2. CHEMICAL ANALYSIS OF BORON CARBIDE POWDER\*

Constituent	Weight Percent
B	75.03
C	19.67
Si	0.1 - 0.3
Ti	0.05 - 0.1
Fe	0.03 - 0.04
Al	0.01 - 0.03
Ca	0.01 - 0.02
Ba	0.005 - 0.01
Mg	0.001 - 0.002

\*Analysis performed by Walker Chemicals, New York

## B. Fabrication

Fabrication was carried out by a variety of processing techniques to establish the most cost-effective process technology. In all, five processes were explored: hot pressing, impulse resistance sintering, cold forming and liquid-phase sintering, induction melting, and arc melting.

### 1. Hot Pressing

Standard vacuum hot-pressing procedures were utilized. Sufficient mixed powder (100 to 150 grams) was placed into a 4-inch-diameter graphite die lined with 0.015-inch-thick Grafoil to produce hot-pressed disks about 0.25-inch thick. After preliminary evacuation, the temperature was raised at the rate of 10 to 15 C/minute and at a preselected temperature, a pressure of 4000 to 5000 psi was applied and held for a period of 15 to 60 minutes. After sintering, the pressure was released and the specimens were slowly furnace cooled in the die. Density data for the sintered specimens were obtained by measuring the density of the individual specimens by a water displacement technique. Densification data, which defined processing parameters with respect to time, temperature, and pressure cycle, were used primarily for process characterization rather than for kinetic studies.

### 2. Impulse Resistance Sintering

Impulse resistance sintering has been the object of considerable interest at AMMRC as a promising method for ceramic fabrication.<sup>7</sup> In this method, 20 to 30 grams of mixed powder was maintained under a constant pressure of 2000 psi in a 2-inch graphite die assembly heated by passing a high electrical current (varying between 2000 and 2800 amperes) directly through the powders. The heating period was between 5 to 12 minutes, as compared to hours, for consolidation by conventional hot pressing. Exact temperature measurement of the compacts was not possible because of reaction with the thermocouple. Therefore, in all experiments the die temperatures were measured, varying between 1000 to 1200 C.

7. SHEPARD, L. A., and CROFT, W. J. *Impulse Resistance Sintering*. Army Materials and Mechanics Research Center, AMMRC TR 72-37, December 1972.

### 3. Cold Forming and Liquid-Phase Sintering

For conventional pressureless sintering, mixed compositions of silicon and 10 to 20 wt%  $B_4C$  were cold pressed into a 1-inch steel die at a pressure varying between 3000 and 7000 psi. The compacts were placed inside a Grafoil-lined graphite crucible, covered with a graphite plug for insulation. Sintering was carried out in a cold-wall vacuum furnace at temperatures ranging between 1200 to 1500 C, with a vacuum between 2 to 10 microns, for a period of 15 to 60 minutes.

After promising compositions were selected, based on microstructures, properties, and ease of fabrication, scale-up to 4-inch-diameter disks was carried out. For scale-up operation, the powder was warm pressed at a temperature of 600 to 800 C and a pressure of 2000 to 3000 psi. The compacts were subsequently sintered in the furnace within a Grafoil-lined graphite liner floating freely from the water-cooled base plate of the furnace, and sintered at temperatures of 1330 to 1550 C for a period of 20 to 60 minutes.

In an attempt to further reduce the potential overall process costs, 10 to 20 wt% SiC was also added to Si to develop dispersed phase microstructures instead of the more costly  $B_4C$ . The sintering was carried out under identical experimental conditions, i.e., at 1200 to 1500 C for a period of 15 to 60 minutes in vacuum of 2 to 10 microns.

Further, a feasibility study was made to cold form *various* shapes by the slip-casting technique. For this purpose, the composition Si-20 wt%  $B_4C$  was mixed in a rubber-lined jar with 30 wt% distilled water without any deflocculant or organic additive. The slip was cast in standard plaster molds for a period of 30 to 60 minutes and then placed inside an oven for drying at temperatures of 150 to 200 C overnight. The dried bodies were sintered in a cold-walled vacuum furnace at temperatures of 1400 to 1500 C for 20 to 30 minutes.

### 4. Induction Melting

A graphite crucible was loaded with 3 lb of mixed powder (Si-20 wt%  $B_4C$ ) in the form of 2-inch cold-pressed pellets. The crucible was charged to melt the pellets in vacuum atmosphere (10 to 15 microns); however, an argon atmosphere was introduced as soon as melting was complete. The furnace door was opened and the melt was poured into a preheated graphite mold (4-inch diameter) to prevent cracking of the ingot during cooling.

### 5. Arc Melting

The arc melter consists of a movable tungsten electrode and water-cooled copper mold mounted in an argon-filled chamber. Four to five batches of 200 grams of Si-20 wt%  $B_4C$  mixed powder in the form of 2-inch pellets were charged into the arc melter, double melted and then cast into blocks 1-3/4 inches on a side.



## C. Property Tests

It has been established by Wilkins et al.<sup>8</sup> that density, hardness, and elastic modulus are critical determinants of ballistic performance. The above factors should be evaluated thoroughly and carefully to understand and develop substantially improved armor materials. These are the first steps toward developing hardfacing materials. Therefore, microhardness measurements on the fabricated specimens (Si-10 wt% and Si-20 wt% B<sub>4</sub>C) were made at room temperature on a Tukon tester machine with a Knoop diamond pyramid indenter using a load of 100 grams.

To determine elastic modulus, test bars were machined from 4-inch-diameter disks, fabricated both by hot pressing and pressureless sintering. The test bars had a dimension of 1.75" x 0.250" x 0.125" and were ground on 220-grit diamond wheel with a final surface roughness of 25 to 30 microinches rms. The test bars were subjected to four-point loading (1-inch span), and all testing was performed at room temperature with a standard Instron machine with a crosshead velocity of 0.002 inch/minute.

## RESULTS OF VARIOUS PROCESSING PROCEDURES

### A. Fabrication

#### 1. Hot Pressing

To establish the optimum processing conditions, various compacts of Si, Si-10 wt% B<sub>4</sub>C, and Si-20 wt% B<sub>4</sub>C compositions were hot pressed at temperatures of 1300 to 1450 C for a period of 15 to 60 minutes; the results are listed in Table 3. The

Table 3. HOT PRESSING OF Si AND Si-B<sub>4</sub>C COMPOSITIONS

Composition	Temperature (deg C)	Pressure (psi)	Time (Hr)	Relative Density (%)
Silicon	1300	4000	1	88.0
	1360	4000	1/2	92.0
	1380	4000	1/2	92.0
	1340	4000	1/2	91.4
	1360	4000	1	92.0
	1380	5000	1	*
Si-10 wt% B <sub>4</sub> C	1340	5000	3/4	97.0
	1380	5000	1	†
	1340	5000	1/2	95.3
	1370	5000	1	99.8
	1360	5000	1	99.7
Si-20 wt% B <sub>4</sub> C	1370	5000	1	90.0
	1460	5000	1/2	‡
	1420	5000	1/2	98.6
	1440	5000	1/2	99.4
	1440	5000	1	99.7

\*Stuck to the die

†Melted and stuck to the die and cracked

‡Melted and reacted with the die

8. WILKINS, M. L. *Third Progress Report of Light Armor Program*. Lawrence Radiation Laboratory, Livermore, Report VCRL-50460, July 1968.

process parameters for fabrication of high density Si-10 wt% B<sub>4</sub>C composition are established to be 1370±10 C at a pressure of 5000 psi for a period of 30 minutes, while parameters for Si-20 wt% B<sub>4</sub>C composition are 1440±10 C at 5000 psi for 30 minutes. Figure 1 shows hot-pressed microstructures of Si with 10 and 20 wt% B<sub>4</sub>C additions.

By contrast, a high final density (>92 to 93%) could not be achieved by hot-pressing Si, without an addition of B<sub>4</sub>C due to the simultaneous secondary grain growth and pore growth which is shown in Figure 2.

## 2. Impulse Resistance Sintering

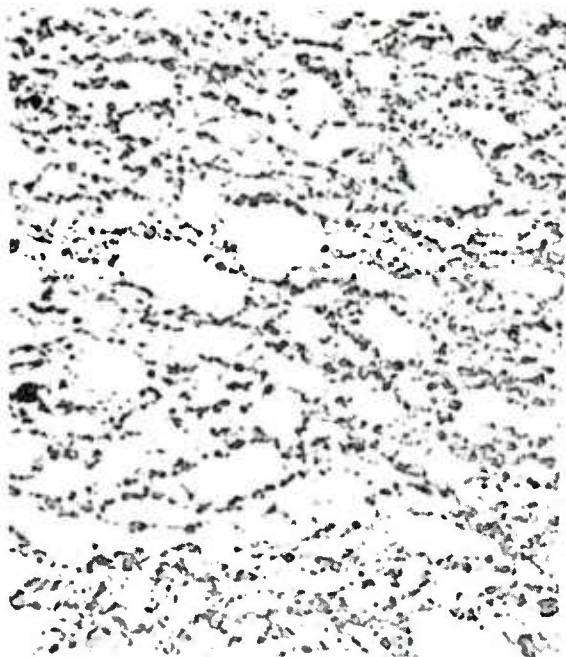
The feasibility of impulse resistance sintering was examined by fabricating 2-inch-diameter specimens. The process can be utilized with carefully controlled conditions to achieve high final density bodies of Si-B<sub>4</sub>C compositions; the results are listed in Table 4. A typical microstructure developed by this technique is shown in Figure 3. However, cracking in the finished specimens was a major problem due to very fast cooling. Also, a density gradient in the sample from the center to the edge was observed due to nonuniform, horizontal current densities, through the powder particles. It is apparent that this effect would be more pronounced in larger specimens during scaling-up operations. Therefore, further work on this fabrication technique was terminated.

## 3. Cold Forming and Liquid-Phase Sintering

Both hot pressing and impulse resistance sintering are batch processes, and are not expected to be cost effective when compared to the pressureless continuous sintering process. (The present state-of-the-art in continuous hot-pressing technique is described in the Appendix.) Therefore, sintering without pressure was carried out at temperatures ranging between 1400 and 1550 C where densification proceeds by liquid-phase sintering. The sintering technique was successful and the process parameters are established to be 1410 C for Si-10 wt% and 1520 C for Si-20 wt% B<sub>4</sub>C, with a sintering time of 30 minutes. The results are shown in Table 5. A unique dispersed phase microstructure has been produced by sintering in the presence of a liquid phase as shown in Figure 4. Phase identification in the sintered specimens was carried out by metallography, X-ray diffraction, and electron probe analyses. X-ray diffraction patterns of as-received Si and B<sub>4</sub>C powders were compared with Si-10 wt% B<sub>4</sub>C and Si-20 wt% B<sub>4</sub>C compositions. In specimens sintered for a short time, major phases were Si and B<sub>4</sub>C with a minor

Table 4. IMPULSE RESISTANCE SINTERING OF  
Si-20 wt% B<sub>4</sub>C COMPOSITIONS

Approx. Temperature (deg C)	Pressure (psi)	Time (min)	Power (amp)	Relative Density (%)
1000	1000	6	2000	75.0
1000	2000	6	2000	84.0
1200	2000	7	2600	94.5
1200	2000	8	2600	95.3
1200	2000	12	2600	98.8
1200	2000	12	2200	88.4
1200	2400	12	2400	92.6



(a) Si-10 wt% B<sub>4</sub>C



(b) Si-20 wt% B<sub>4</sub>C

Figure 1. Microstructures of hot-pressed Si-B<sub>4</sub>C compositions. Mag. 500X



Figure 2. Microstructure of hot-pressed silicon. Mag. 500X

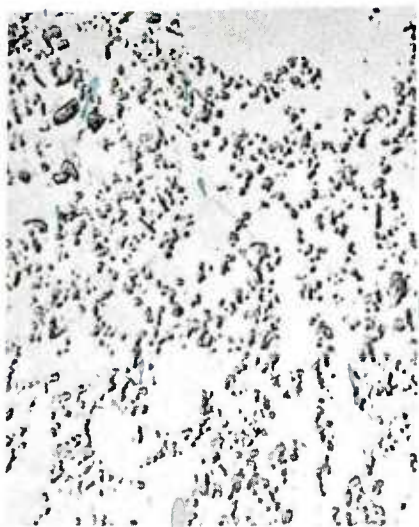


Figure 3. Microstructure of impulse resistance sintered Si-20 wt% B<sub>4</sub>C. Mag. 500X



Table 5. LIQUID-PHASE SINTERING OF Si-B<sub>4</sub>C COMPOSITIONS

Composition (wt%)	Temperature (deg C)	Time (hr)	Remarks
Si-10% B <sub>4</sub> C	1180	1	Poorly sintered
	1520	1	Completely melted
	1280	2	Dense (relative density 95%)
	1380	2	Fully dense and some melting with shrinkage holes
	1410	1-1/2	Fully dense but melted with considerable shape change with shrinkage holes
	1430	3/4	Melted with large shrinkage holes on the top of the compact
	1380	1	Fully dense with glazed surfaces indicating some melting. No shape change occurred
	1380	1/2	Not fully dense (97.6%) with dull surfaces. No shape change
	1410	1/2	Fully dense and best surface finished compact
	1430	1/2	Fully dense but some melting with shape change
Si-20% B <sub>4</sub> C	1520	1	Fully dense with glazed surfaces and no shape change
	1430	1	Dense but porous at the center forming a lens shape
	1420	2	Not fully dense (92%), but no lens effect due to longer sintering
	1370	2	Very poorly sintered
	1500	1	Fully dense with light-glazed surfaces
	1520	1/2	Fully dense with light-glazed surfaces
	1520	3/4	Fully dense with medium-glazed surfaces

(a) Si-10 wt% B<sub>4</sub>C(b) Si-20 wt% B<sub>4</sub>CFigure 4. Microstructures of liquid-phase sintered Si-B<sub>4</sub>C compositions. Mag. 400X

amount of SiC phase; in the specimens sintered for longer times (30 to 120 minutes), SiB<sub>4</sub> was also detected. However, considerable shape changes were found to occur in the finished specimens containing 10 wt% B<sub>4</sub>C due to large volume shrinkage during cooling. This effect was further enlarged during scaling up to 4-inch-diameter size and turned out to be a critical factor in fabrication of large sizes and complex shapes.

By contrast, the shape change in the Si-20 wt% B<sub>4</sub>C composition was minimized due to increased B<sub>4</sub>C content. This composition was found to be more suitable for scale-up operation and subsequently pursued for ballistic evaluation.

To make the overall process more cost effective, SiC (less expensive than B<sub>4</sub>C) was used with Si to develop dispersed phase microstructures. Figure 5 shows a typical microstructure of such compositions. Segregation of SiC particles was observed during liquid-phase sintering which was attributed to the nonwetting tendency of Si to SiC (Figure 5). Therefore, the scale-up fabrication of this composition was discontinued.

A further attempt was made to fabricate a dispersed phase composition utilizing Si and C with a preselected proportion to synthesize SiC compound dispersed in Si matrix. Figure 6 shows such a microstructure, where SiC grains have been formed during sintering by reaction with Si and C. This composition has good potential because of cheaper raw materials sources, however, the finished items are heavier than the Si-B<sub>4</sub>C dispersion.

The feasibility study of other cold-forming processes, such as slip casting, has been successfully demonstrated in this investigation to produce complex shapes. Figure 7a shows a 4-inch radome cold formed by the slip casting process. Also, 3" x 3" flat plates (Figure 7b) and crucibles have been cold formed successfully by slip casting of Si-20 wt% B<sub>4</sub>C composition.

#### 4. Induction Melting

Induction melting was carried out to fabricate 4" x 6" ingots. One of the important questions was whether the melting process retains a uniform dispersion in the resulting microstructures or leads to localized segregation of B<sub>4</sub>C particles during melting of Si-20 wt% B<sub>4</sub>C composition. The process was unsuccessful because of the inability of the furnace, even at its maximum operating capacity, to melt the composition uniformly, therefore, this approach was dropped.

#### 5. Arc Melting

Arc melting produced a change in localized composition resulting in a non-homogeneous microstructure. Figure 8 shows segregated microstructures formed during arc melting. Electron probe analyses indicated that the large gray angular phase has the composition of SiB (Figure 8a), while the dark gray lath-shaped (pitted) phase is identified as SiC (Figure 8b). Due to very high operating temperatures, SiB<sub>4</sub> and SiC compounds were predominant. However, thermal stresses were very high due to fast cooling, resulting in cracking in all specimens. As a consequence this approach was also dropped.



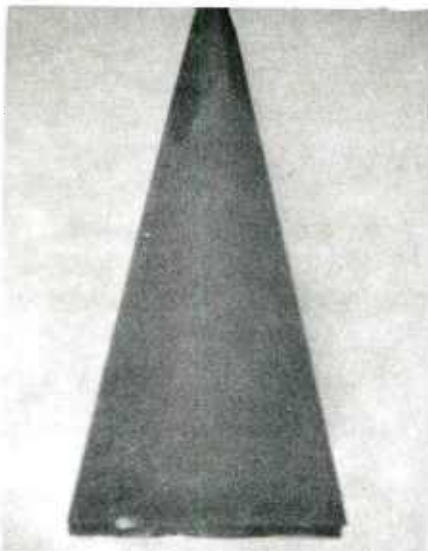


Figure 5. Microstructure of Si-10 wt% SiC by liquid-phase sintering. Mag. 500X

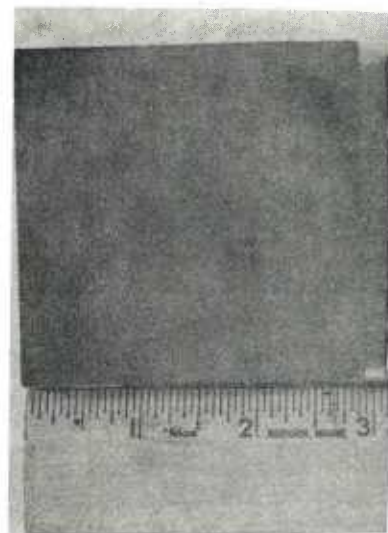


Figure 6. Microstructure of Si-5 wt% C by liquid-phase sintering. Mag. 500X

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(a) Slip-cast radome (4")



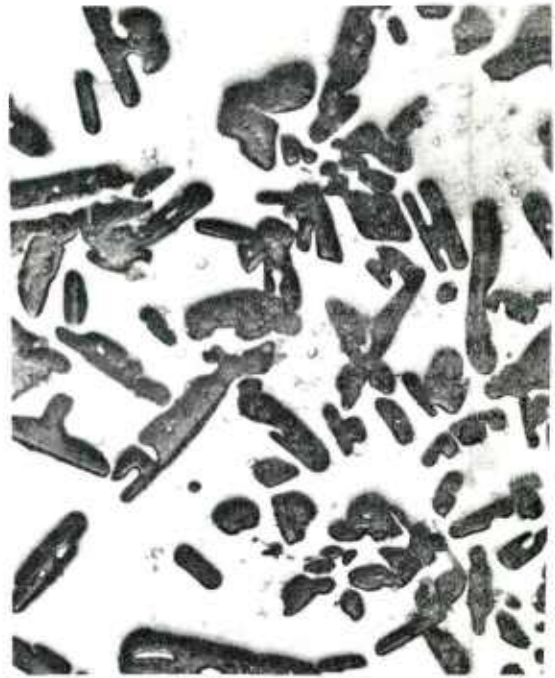
(b) Slip-cast flat plate (3" x 3" x 3/8")

Figure 7. Cold forming of Si-20 wt% B<sub>4</sub>C compositions into complex shapes by slip-casting technique.

19-066-1114/AMC-75



(a) Showing formation of  $\text{SiB}_4$  phase



(b) Showing formation of  $\text{SiC}$  phase

Figure 8. Microstructures of Si-10 wt%  $\text{B}_4\text{C}$  by arc melting. Mag. 500X

19-066-1216/AMC-75

## B. Property Evaluation and Fracture Characteristics

The microhardness was determined by a diamond Knoop indenter with 100-grams load. The average microhardness of single-phase Si was around 800 to 900  $\text{kg/mm}^2$ . The addition of 10 wt%  $\text{B}_4\text{C}$  increased the average hardness to around 1300 to 1440  $\text{kg/mm}^2$  while 20 wt%  $\text{B}_4\text{C}$  addition yielded an average of 1600 to 1700  $\text{kg/mm}^2$ , approximately double the hardness of single-phase silicon. It is apparent, therefore, that the increase in hardness is due to increase in  $\text{B}_4\text{C}$  content dispersed in the Si matrix.

Modulus of rupture (MOR) tests were performed with four-point loading fixtures at room temperature and the results are shown in Table 6. The modulus of elasticity was calculated from load versus strain measurements obtained during the bend tests. Strain measurements were obtained by attaching SR-4 strain gages (type FAP) to the tensile surface of the bend specimens. The data indicates that the modulus of elasticity for Si-10 wt%  $\text{B}_4\text{C}$  varies between 26 to 29  $\times 10^6$  psi, while for Si-20 wt%  $\text{B}_4\text{C}$ , the modulus of elasticity is 33 to 38  $\times 10^6$  psi. It was found that specimens fabricated by solid state sintering have higher MOR than that produced with liquid-phase sintering or melting. The net effect, however, was a threefold increase in the MOR value with a  $\text{B}_4\text{C}$  addition of 20% over that of single-phase silicon.

The fracture surfaces of transverse bend bars were examined by scanning electron microscopy. Essentially, a mixed intergranular fracture associated with a river pattern was observed as shown in Figure 9. Fractographic investigation of Si- $\text{B}_4\text{C}$  bend bars indicated that fracture initiation in these materials could

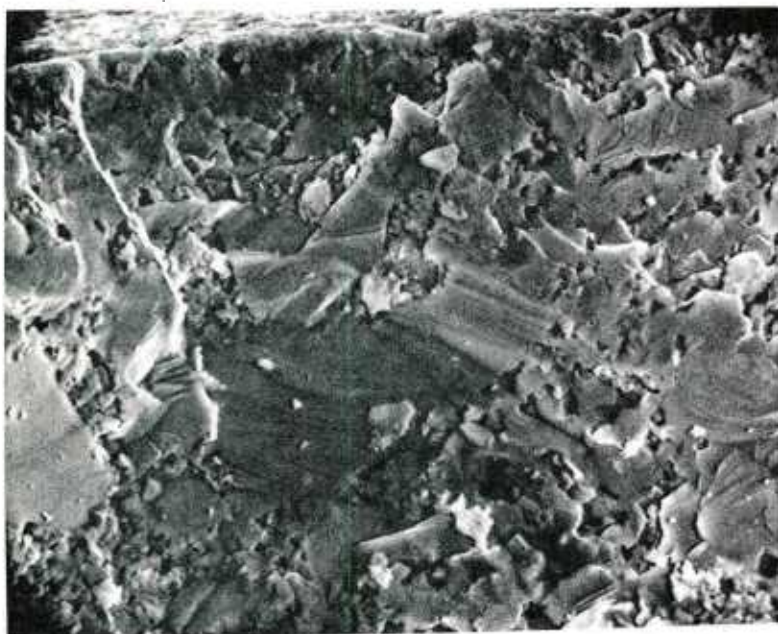
Table 6. FOUR-POINT MODULUS OF RUPTURE STRENGTH OF Si-B<sub>4</sub>C COMPOSITIONS

Specimen	Silicon Hot Pressed (psi)	Si-10 wt% B <sub>4</sub> C Hot Pressed (psi)	Si-10 wt% B <sub>4</sub> C Liquid-Phase Sintering (psi)	Si-20 wt% B <sub>4</sub> C Liquid-Phase Sintering (psi)
1	14,200	16,100	10,400	24,600
2	12,600	21,300	8,000*	24,400
3	12,500	25,600	10,600	19,000†
4	8,300‡	25,900	11,200	25,100
5	13,300	24,200	10,600	26,500
6	11,000	22,400	9,800	31,300
Mean MOR	11,983	22,583	10,100	25,150
Elastic Modulus	20 x 10 <sup>6</sup>	-	26-29 x 10 <sup>6</sup>	33-38 x 10 <sup>6</sup>

\*Bar shown in Figure 14

†Bar shown in Figure 12

‡Bar shown in Figure 13

Figure 9. Sem fractograph of Si-20 wt% B<sub>4</sub>C bend bar, showing mixed intergranular and transgranular fracture associated with river pattern. Mag. 700X

19-066-1216/AMC-75

be traced to localized large pores, on the order of several grains in diameter, near the tension surface of the bend specimens. This was particularly true in specimens which failed at inordinately low stresses. For example, Figure 10 shows the fracture pattern of a bend bar which failed at very low stress of 8000 psi. The fractograph shows localized and isolated pore pockets which initiated fracture. In some cases, large agglomerated grains surrounded by small grains contributed to fracture initiation at low stress level, as shown in Figure 11, in which the bend bar failed at a stress level of 8300 psi. This observation shows that microstructural inhomogeneities, pores or grains or clusters thereof, seem to be a major source of strength degradation and confirms the work reported by Rice,<sup>9</sup> that microstructural dependence must be correlated with the character of actual failure origins in order to understand the strength behavior of ceramic materials.

9. RICE, R. *Fractographic Identification of Strength-Controlling Flaws and Microstructure in Fracture Mechanics of Ceramics* Volume 2, R. C. Bradt, D. P. H. Hasselman, and F. F. Long, ed., Plenum Press, New York, 1974, p. 323.





Figure 10. Fracture pattern of Si-20 wt% B<sub>4</sub>C, indicating isolated pore pockets as failure origin. The bend bar failed at 8000 psi (MOR). Mag. 700X  
19-066-1215/AMC-75



Figure 11. Fracture pattern of hot-pressed Si, indicating large agglomerated grains associated with surrounding small grains as failure origin. The bend bar failed at 8300 psi (MOR). Mag. 700X  
19-066-1215/AMC-75

## CONCLUSIONS

1. Si-B<sub>4</sub>C compositions containing 10 to 20 wt% B<sub>4</sub>C were fabricated by using various processing techniques such as hot pressing, impulse resistance sintering, liquid-phase sintering, induction melting, and arc melting. Among all the techniques investigated, hot-pressing and liquid-phase sintering are most promising with respect to density, microstructures, ease of fabrication, and yield rate.
2. The hot-pressing parameters for Si-10 wt% B<sub>4</sub>C and Si-20 wt% B<sub>4</sub>C are established to be 1370±10 C and 1440±10 C, while the processing parameters by liquid-phase sintering technique are 1410±10 C and 1520±10 C.
3. The resulting compositions produced dispersed-phase microstructures containing B<sub>4</sub>C particles in Si matrix.
4. The average microhardness of Si-10 wt% B<sub>4</sub>C composition is determined to be 1300 to 1440 kg/mm<sup>2</sup> with modulus of elasticity of 26 to 29 x 10<sup>6</sup> psi, while for Si-20 wt% B<sub>4</sub>C compositions, the microhardness is 1600 to 1700 kg/mm<sup>2</sup>, with an elastic modulus of 33 to 38 x 10<sup>6</sup> psi.
5. The operation was scaled up to produce 4-inch-diameter disks; the most appropriate composition for scale-up was found to be Si-20 wt% B<sub>4</sub>C due to minimum shape changes in the finished specimens.
6. Cold forming of flat plates and complex shapes (radomes, crucibles) was successfully achieved by the slip casting process.
7. A sufficient number of plates (4-inch diameter) produced by the fabrication processes discussed in this report have been subjected to ballistic testing, the results of which will be presented later.

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## APPENDIX. PRESENT STATE-OF-THE-ART IN CONTINUOUS HOT PRESSING

Hot pressing has been essentially a batch process, although various schemes have been proposed to make the process more economical. Die assemblies maintained hot with a heated feed and hot ejection have been suggested, but there is no evidence that such a system has been tried.\* The use of many die assemblies that are preheated, passed under a press, and then cooled, has been proposed,† but die costs appear to be a prohibitive factor. Stacked pressings of a number of pieces in a single die and gang pressing of a number of die assemblies have both been used successfully. In particular, stacked pressing is the only semicontinuous technique which is now in common practice for commercial production by various companies such as: Norton Company, Avco Corporation, Ceradyne, Fiber Materials Incorporated, Boride Products, etc.

In addition, however, sequential pressing of a number of specimens has been used in a horizontal hot press on a semicontinuous basis. Here, one die setup is charged from one end to the hot zone, where a preselected total pressure is applied and held for a desired time period for densification. However, it has been found that the apparent cost of operating this type of horizontal setup is higher due to the following reasons:

1. More personnel are required intermittently for an entire eight-hour period for loading, unloading, and aligning for each charge setup.
2. Since final densification occurs by diffusional processes, an optimum time is required for each batch under temperature and pressure before unloading from the other end. This reduces the productivity per unit time.
3. Rapid unloading increases the scrap rates due to cooling cracks.

By contrast, in a standard vertical hot press, multiple stacking of cold prepressed tiles is common practice, and twelve to fourteen tiles (6" x 6") have been hot pressed in one single run. Once the tiles are loaded, only one person is required for the control of the operation compared to three or four persons in a horizontal setup. Moreover, it would be difficult to fabricate large size and contour shapes in a continuous horizontal apparatus.

In addition to the foregoing, the efforts of Oudemans‡ and others toward automatic and/or continuous hot-pressing processes should play an important role in the greater use of hot pressing. It is inevitable, however, that although the hot-pressing process can be made continuous for very small samples, or simple shapes, it would be difficult for larger sizes and shapes and the unit costs will be high. More extensive furnace design work is essential in the area before any substantial improvement in cost effectiveness can be achieved.

\*Committee on Ceramic Processing. *Ceramic Processing*. National Academy of Sciences Publ. 1576, 1958, p. 38.

†FULRATH, R. M. *Critical Compilation of Ceramic Forming Methods*. U. S. Air Force Materials Laboratory Tech. Doc. Rept. No. RDT-TDR-63-4069, 1964, p. 33. AD 431002.

‡Oudemans, G. J. *Continuous Hot Pressing*. Phillips Technical Review, v. 29, no. 2, 1968, p. 45-53 also, Proc. Brit. Ceram. Soc., v. 12, no. 83, 1969.

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BASED COMPOSITES FOR ARMOR APPLICATIONS -  
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